Fatigue Failure Mechanisms of Mg-Y Dilute Alloys Based on Dislocation Plasticity

著者	HUO QINGHUAN
number	62
学位授与機関	Tohoku University
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氏　　　　　名	ふぉ　しんはん HUO　QINGHUAN
授　与　学　位	博士（工学）
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指　導　教　員	東北大学教授　小池　淳一
論文審査委員	主査　東北大学教授　小池　淳一 東北大学教授　千葉　晶彦　　東北大学教授　吉見　享佑

論文内容要旨

Magnesium (Mg) alloys, known as the lightest structural materials, have attracted considerable interests in the transportation and aerospace industry due to their low density, high specific strength and good castability. However, their poor mechanical properties and low creep resistance, resulting from their hexagonal close-packed (HCP) structure and insufficient number of slip systems, have limited many other potential applications. It was demonstrated that their mechanical properties at ambient temperature could be improved by adding rare earth (RE) elements to Mg alloys. Among the Mg-RE alloys, Mg-Y binary alloy is one good series. Due to the relatively low price of yttrium (Y) in comparison to other RE elements, Mg-Y alloys have been proved to have good ductility at room temperature. Besides undertaken static loading, Mg-Y alloys are also requested to serve as a structural material under cyclic loading. Thus, the fatigue failure mechanisms are the key factors on the fatigue behavior of Mg-Y alloys. To investigate the specific fatigue failure process, it is important to know the dislocation plasticity before crack initiation. With the analysis of dislocation plasticity during fatigue cycles, the origin of fatigue failure in Mg-Y alloys can be revealed and the methods of enhancing the fatigue properties can be put forward. Mg-Y alloy has been proved that it has good combination of strength and ductility at room temperature. However, the fatigue properties and corresponding fatigue mechanisms are still few investigated. Whether good ductility can bring high fatigue limit? It remains unknown. Up to now, there is no report involving the fatigue mechanisms of Mg-Y alloy. It needs to be studied in detail. Besides, most published investigations about the fatigue of Mg alloys were concentrated on the crack propagation and failure rate. There are still few reports involving the basic mechanisms or initial origins of fatigue failure. It is important to know how the fatigue failure happens. That can promote the understanding of the fatigue of Mg alloys.

The samples used in this study were hot-extruded sheets of pure Mg, Mg-0.06at%Y, Mg-0.6at%Y alloy sheets and hot-rolled Mg-1.0at%Y and AZ31 alloy sheets. For briefness, the samples are named pure Mg, 006Y, 06Y, 1Y and AZ31 hereafter. The maximum content of Y atom for no precipitates is about 2.2 at%. Thus, the Mg-Y alloys used in this work have no precipitate interior the grains or along the grain boundaries after homogenization annealing. Dog-bone shaped samples were prepared by cutting the sheets with an electro discharge machine. The dimension of gauge area was 10 mm in length (parallel to the rolling direction, RD), 3.6 mm in width (parallel to the transverse direction, TD), and 1.8 mm in thickness (parallel to the normal direction, ND). Since twinning is easily activated in large grains above 20 μm but hardly activated in fine grains below 20 μm, homogenization treatments were performed under different conditions. All the obtained samples had equiaxed grains with fine or large average grain sizes. In the first case, 006Y, 06Y, 1Y and AZ31 alloys were annealed at relatively low temperatures to get fine grains below 10 μm. For pure Mg, due to no segments existing along the grain boundaries, it is too difficult to obtain fine grains below 10 μm. In the second case, all the alloys with different contents were annealed at relatively high temperatures to obtain coarse grains. Their average grain sizes were all above 180 μm. In order to identify dominant deformation mechanisms from slip traces, a mirror finished surface was prepared by the following method. The surfaces of the samples were mechanically ground with #1200, #2400, and #4000 abrasive papers, followed by polishing with 1 and 0.25 μm diamond pastes. Finally, chemical polishing was carried out using a solution of nitric acid (10 ml) and absolute ethanol (100 ml). These samples were subjected to uniaxial tensile test at room temperature at a strain rate of 1×10^{-3} s^{-1}. The yield stress was measured as the 0.2% proof stress. All the values of the yield stress (YS), ultimate tensile stress (UTS), and fracture elongation (FE) were tested at least thrice. And average values were calculated and used to the stress-strain curves in the results and discussion. Fatigue test was controlled by stress. That means the fatigue test in this work was conventional high-cycle fatigue test. Stress-controlled fatigue test was performed at room temperature with a frequency of 10 Hz. The applied stress was controlled by sinusoidal waveform. The stress ratio was set to $R = 0.1$. That is to say, the ratio of the minimum stress value by the maximum stress value is 0.1. Cyclic tensile test was carried out at room temperature at a strain rate of 1×10^{-3} s^{-1} for 10 cycles by continuously changing applied stress between zero and peak stress, σ_{max}. The peak stress values were selected based on YS obtained from uniaxial tensile test: below YS, nearly equal to YS and above YS. Through cyclic tensile test, important parameters could be obtained to analyze the specific fatigue mechanisms of Mg-Y alloys. After mechanical testing, the gauge areas of the tested samples were cut down for surface and cross-section observations. Surface observations were performed by using an optical microscopy (OM) and a scanning electron microscopy (SEM). Also, the slip traces on the surface were observed by a focused ion beam (FIB) microscopy. The internal microstructures of the cross-sections were observed by a transmission electron microscopy (TEM). The TEM samples were prepared with the assistance of FIB. By using FIB observation, the interested areas could be easily found and the TEM samples could be directly made from the certain areas without too much damage on the sample surfaces. The crystallographic orientation distributions were checked by an electron backscatter diffraction (EBSD) apparatus. It is noteworthy to state here that no more polishing would be subjected to the sample surfaces. In this way, all the information of slip traces on the surfaces can be well remained during the SEM and FIB observations. In other words, one sample is only polished once. The polishing is only done before its mechanical experiments.

In the case of fine grains, AZ31 alloy has the highest fatigue limit, followed by 1.0Y, 0.6Y and 0.06Y alloys. The fatigue limit of all tested samples is between microyielding stress and macroyielding stress. There is a good relation between fatigue limit and yield stress. Higher yield stress corresponds to higher fatigue limit. Also, one equation of $\Delta\varepsilon_p = - \beta \bullet \log N + C$ can be used to evaluate the cyclic strain hardening. The constant β represents the speed of strain hardening and it can be used as a parameter to evaluate fatigue limit. Higher β value corresponds to lower fatigue limit. Only the basal slip occurs below fatigue limit while the non-basal slip occurs above fatigue limit. The non-basal slip is the origin of fatigue failure in Mg alloys with fine grains. The higher activity for the non-basal slip induces the early fatigue failure in Mg-Y alloys.

In the case of large grains, AZ31 has the highest fatigue limit, followed by 1.0Y, 0.6Y, 0.06Y and pure Mg. The fatigue limit of all tested samples is below their corresponding macroyielding stress. Although the relations between fatigue limit and mechanical properties are roughly matched each other, the relations are not in a linear behavior. Inversely, the equation of $\Delta\varepsilon_p = -\beta \bullet \log N + C$ can be still applied to evaluate the fatigue properties. The β constant obtained from cyclic tensile test has an excellent relation with fatigue limit. Higher β constant corresponds to lower fatigue limit. Only the basal slip occurs below fatigue limit while the non-basal slip occurs above fatigue limit. The non-basal slip is the origin of fatigue failure in Mg-Y series while double twinning is the origin of basal textured AZ31 alloy.

Although the CRSS of non-basal slip is decreased in Mg-Y alloys, even the CRSS of prismatic slip is possibly reduced to below the CRSS of {10-12} twinning, residual twins are still largely found. As the resolved shear stress for detwinning is lower than activating new dislocations, detwinning is easily complete at first. As the cycle number increasing and deformation becoming heavier, detwinning behavior is harder. New dislocations will be activated and move towards twin boundaries to impede the complete detwinning. As a result, the number of residual twins is increased. When the cycle number is further increased or the stress level is higher, the high-density dislocations can impede more twins from detwinning.

In Mg alloys, there is real fatigue limit existing in Mg alloys during fatigue cycles. And the S-N curve has a prominent bending point around 10^5 cycles. Based on the classical definition of fatigue test, the number of cycle below 10^5 cycles is called low-cycle fatigue while the number of cycle above 10^5 cycles is called high-cycle fatigue. The bending point always occurs at the critical point between low-cycle fatigue and high-cycle fatigue. The real fatigue limit means that if further cycle is subjected to Mg alloys after 10^7 cycles, there would be still no fatigue crack initiation. The reason can be explained as follows. In the period of low-cycle fatigue, as the results and discussion about pyramidal slip in the present study, there would be plenty of pyramidal slip happening in the Mg-Y alloy samples. The pyramidal slip is the origin of fatigue failure in Mg-Y alloy under high stress level. When the stress level is reduced to below fatigue limit, pyramidal slip is hardly to be activated. Only basal slip can be found in the samples. Then there would be no origin of fatigue failure. Mg alloys can continuously undertake the fatigue cycles. Of course, in AZ31 alloy which has a strong basal texture, it has no pyramidal slip in the low-cycle fatigue period. However, double twinning can be found to be the origin of surface step. Double twinning is caused by stress concentration. Stress concentration is caused by the stress concentration in the adjacent grain in which cross-slip is existed. If the stress level is lower than fatigue limit of AZ31 alloy, there would be no cross-slip. Then there would be no double twinning. Therefore, different deformation modes cause different fatigue behaviors in low-cycle fatigue period and high-cycle fatigue period.

The same points between the results of fine-grained alloys and large-grained alloys contain: The fatigue limit of the tested samples is below their corresponding macroyielding stress. The equation of $\Delta\varepsilon_p = -\beta \bullet \log N + C$ can successfully employed to both the cases of fine-grained and large-grained Mg alloys. The constant β has a good relation with fatigue limit in both cases of fine and large grains. Only the basal slip occurs below fatigue limit while non-basal slip occurs above fatigue limit.

The different points between the results of fine-grained alloys and large-grained alloys contain: Grain refinement can improve fatigue limit. In fine grains case, the relation between β and fatigue limit is lying on the same slope, due to the non-basal being easily activated in fine grains. In large grains case, higher activity for the non-basal slip leads to more serious strain hardening in Mg-Y alloys. The pyramidal slip is the origin of fatigue failure in Mg-Y alloys while double twinning is the origin in AZ31 alloy.

The novel points of this work contain: 1) Although pyramidal slip is treated as a positive deformation mode under uniaxial loading based on many previous reports, pyramidal slip is found to be the origin of fatigue failure of Mg-Y alloy. To serve as a structural material, the pyramidal slip of Mg-Y dilute alloy should be paid attention in the future application. 2) Although weak basal textured Mg-RE alloys are of better plasticity compared to strong basal textured Mg alloys, the high activity for pyramidal slip increases the possibility of serious strain hardening during fatigue cycles. And the enhanced strain hardening leads to unexpected fatigue failure of Mg-RE alloys. 3) Although the yield stress or ultimate tensile strength is used to predict the fatigue limit in most structural metals, it is lack of the consideration of grain size and texture. In the present work, it is the first time found that the β constant for cyclic strain hardening can match fatigue limit very well in both cases of fine and large grains and in both cases of strong and weak texture.

In summary, although the non-basal slip promotes the ductility of Mg-Y alloys under static loading, the non-basal slip can induce heavy strain hardening. With the fatigue cycle number increasing, the cyclic strain hardening is accumulated and finally leads to the fatigue failure. It is suggested that the non-basal slip should be considered as the key factor during the application of Mg-Y alloys under cyclic loading or fatigue cycles.

論文審査結果の要旨

マグネシウム（Mg）合金は、軽量構造材料として注目されているが、室温での延性が乏しく機械加工が困難な材料である。しかし、微量のイットリウム（Y）を添加することによって室温延性が改善されることから、Mg-Y 合金の変形機構に関する多数の報告がなされている。一方で、実用に供する際には、繰り返し荷重を付加した際の、疲労変形・破壊挙動を知ることが重要である。本研究は、疲労破壊に至る前段階の繰り返し変形挙動に着目して、Mg-Y 合金の疲労強度と疲労変形の特徴を明らかにし、高延性を発現するメカニズムと疲労変形メカニズムの関係を明らかにすることを目的とした。

実験に用いたサンプルは、Mg – 0.06, 0.6, 1.0 at.%Y 合金の押し出し材あるいは圧延材とした。また、比較試料として AZ31 合金の圧延材を用いた。熱処理を行って、結晶粒径が 200 μｍの粗大粒試料と 10 μｍの微細粒試料を作製した。これらの試料に対して、室温における定速引張試験、繰り返し変形試験、疲労試験を実施した。微細組織、結晶方位分布、すべり線の観察を走査型電子顕微鏡（ＳＥＭ）で行い、転位のすべり系の同定を透過型電子顕微鏡（ＴＥＭ）を用いて行った。

粗大粒試料の定速引張試験を実施し、変形試料の内部組織をＴＥＭで観察したところ、0.2%耐力近傍において柱面転位と錐面転位が活動していることが判明した。ＳＥＭによる試料表面のすべり線の観察においても同様の結果が得られた。Ｙ添加によって錘面転位の活動が活発になり、その結果、延性が向上することが明らかになった。疲労強度を測定したところ、0.2%耐力にほぼ等しい応力においてＳ－Ｎ曲線に変曲点が見られた。通常はこの応力以下では疲労破壊が生じず、疲労限とされる。しかし、本試料においては、さらに小さい応力においても疲労破壊が生じ、疲労限が存在しないことを示唆している。定速引張試験を実施するにあたりひずみゲージを装着して応力・ひずみ関係の精密測定を行ったところ、応力負荷開始とほぼ同時に底面転位すべりによるミクロ降伏が発生し、引き続いて双晶形成が確認された。その後、粒界近傍で柱面転位と錐面転位が活動し、マクロ降伏に至った。繰り返し変形試験においては、荷重負荷時に双晶が形成され、除荷時に双晶が消滅することに伴って、応力・ひずみ曲線にヒステリシスが観測された。また、毎回の変形で発生する塑性ひずみが、繰り返し変形回数の増加とともに減少し、加工硬化が生じていることを示した。塑性ひずみと変形回数の対数をプロットすると良好な直線関係が得られ、直線の傾きから硬化係数が得られた。微細粒試料についても上記と同様の実験を行った。粗大粒と異なる点は、ミクロ降伏後に顕著な双晶変形は生じず、繰り返し応力・ひずみ曲線にもマクロ降伏よりかなり大きい応力を付加しないとヒステリシスは観測されない。一方で、柱面転位と錘面転位の活動は観測された。また、Ｓ－Ｎ曲線には明瞭な疲労限が観察された。

得られた結果より、Mg-Y 合金が延性に富む理由は、柱面転位と錘面転位（非底面）の活動が活発化し、独立なすべり系の数が増加したことによる。一方で、非底面転位が活動することによって転位間の相互作用が顕著となり、転位の絡み合い、切り合いによって加工硬化が顕著になる。従って、延性と疲労強度が同じ傾向を示す Al、Cu、Fe などの立方晶金属とは異なり、Mg-Y 合金においては延性を向上する変更機構が加工硬化をもたらし、疲労強度を劣化させることが明らかになった。

以上のように、延性に富むという理由で実用化が期待されている Mg-Y 合金ではあるが、延性と疲労強度が逆の傾向を示すことが初めて明らかになり、Mg-Y 合金の実用化に向けた課題を示すことができた。

よって、本論文は博士(工学)の学位論文として合格と認める。